MOISTURE-TEMPERATURE RELATIONSHIP IN STARCH GELATINIZATION

by

GUSTAVO ENRIQUE PEREZ

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INTRODUCTION

Widespread uses of starch are due mainly to two important properties, film and viscous paste formation. These two characteristic properties have been studied under varied conditions by many research workers. Formation of films and viscous pastes is altered by factors such as H-ion concentration, composition of the starch, structure of the granule, and time and temperature of cooking. These basic factors affect the final starch product in appearance, texture, yield, flavor, and digestibility. This investigation was undertaken to relate moisture and temperature to the gelatinization and pasting properties of various starches.

The effect of percent moisture and degree of heating as related to the percent gelatinization of the starch is a problem faced by manufacturers of starch-containing foods. The work reported here was done to clarify the effect of different moisture levels on starch gelatinization and pasting when subjected to two different temperatures, 100°C. and 200°C.

REVIEW OF LITERATURE

Starch Granule Structure

Raw starch is composed of minute cells or granules so small that it takes three quarters of a trillion to make one pound of corn starch (14). Starch granules from different sources show variety in shape and size and other superficial physical characteristics. The final shape of the starch granule depends to a

large extent on the surrounding environment during its formation (30).

Internally, the granular structure of starch can be explained in terms of the attractive forces between the large carbohydrate molecules. This concept, generally accepted today, was formulated by K. H. Meyer as cited by Leach et al. (35). According to this theory, the intermixed linear and branched starch molecules are arranged in the granule in a radial fashion. Where ever linear segments of either branched or linear molecules parallel one another, hydrogen bonding forces pull the chains together into associated crystalline bundles or micelles. A long linear chain may conceivably pass through a number of such micellar areas while the outer fringe branches of the ramified molecule may participate in several separate micelles. Hence, the crystalline areas hold the granule together, permitting swelling in water at certain temperatures but preventing dispersion and solution of the individual starch molecules. In the spaces between the micelles, the chains and branches are more disordered and hence, less densely packed (35). The starch granule shows the dark cross pattern of a sphero-crystal under the polarizing microscope because the associated bundles of molecules represent an essentially crystalline lattice. The associative forces between the molecules can be overcome if sufficient energy is applied (10). Ponsler (40), in contrast, reported that the regular and uniform arrangement shown by starch when examined under X-rays is destroyed when the granule is crushed, indicating that the regularity is not that of a crystal structure.

As has been indicated, the starch granule is composed of two different fractions, namely the linear amylose and the branched amylopectin, each possessing a different molecular constitution. There has been some confusion about the naming of these two fractions and, furthermore, the existence of amylopectin was doubted for some time. The names alpha- and beta-amylose were used to distinguish the two fractions separated on the basis of their solubility characteristics. The term amylopectin was introduced by Maquenne and Roux (37), in 1905, as the name for the non-saccharifiable portion of starch (32). Samec and Haerdtl (45), in 1920, reported that starches consist of a highly viscous, electro-conducting fraction which precipitates on electrodialysis and a non-viscous, non-electro-conducting fraction which is not precipitated. The amounts vary according to the origin of the starch, their relative proportion influencing the viscosity, resistance to amylase, and water absorption. In 1926, alpha-amylose was considered the equivalent of amylopectin. Later, in 1933, the two terms were used synonymously (32). In 1934, Maquenne and Roux (37) described the concept of starch retrogradation.

The amylopectin molecule is considered to be made up of a large number of branches, each approximately 25 glucose units in length. The branches themselves are combined in such a way that the free reducing groups of the glucose unit of a branch are linked through the sixth hydroxyl group of a glucose in an adjacent branch (alpha 1-6 glucosidic linkage), thus forming a ramified structure. The evidence for a 1-6 linkage in amylopectin

has been well substantiated by the isolation of beta-D-isomaltose as the crystalline acetoacetate from an acid-hydrolyzate of amylopectin (16). The branched portion is thus a mixture of polymers of unbranched chain molecules of varying length (19). Amylopectin appears to contain a small amount of phosphoric acid as part of the molecule. According to Czaja (12), amylopectin is concentrated at the periphery of the granules. Czaja also deduced from the behavior during swelling of the starch granule that amylopectin is mainly responsible for the optical activity of the granule. Amylopectin is more resistant to attack by beta-amylase since the action of this enzyme is limited to the open-end chain of the branched molecule. Amylopectin yields, upon partial acid hydrolysis, various amounts of glucose, maltose, maltotriose, tetraose and pentaose, isomaltose, panose, and nigerose.

Amylose, or the linear fraction, consists of glucose units exclusively united through alpha 1-4 glucosidic bonds. It is hydrolyzed by both alpha- and beta-amylase, giving as products, a mixture of glucose and maltose (54), and in addition, some maltotriose, tetraose, and pentaose. Amylose appears to assume a randomly coiled configuration in solution when it is complexed with iodine, fatty acids, and higher alcohols. The amylose-amylopectin ratio of different sources of starch are known to vary widely although many common cereal starches, such as wheat and corn, usually have a 1:3 amylose-amylopectin ratio (19,20). It must be recognized that the determination of amylose-amylopectin ratios are dependent on the method of separation and that certain

artifacts might be introduced during the separation. Spark (51) found the X-ray diffraction pattern of amyloses depended more on the method of separation than on its source.

The properties of the isolated amylose differ from those of amylopectin in several respects (19,39,50). Amylose is slightly soluble in cold water but is soluble in boiling water to the extent of 7 to 10 percent, and the small amount that dissolves will rapidly retrograde from solution. It is completely hydrolyzed by beta-amylase. It has a great tendency to absorb iodine and produces a sharp, V-type X-ray pattern. There is evidence that amylose possesses a helical structure when complexed with iodine, high molecular weight fatty acids, or alcohols. On the basis of a starch-iodine complex, the blue color, on addition of iodine, may be explained adequately. The iodine molecules are assumed to be deposited within the amylose spiral, forming a complex which produces the blue color.

Amylopectin is quite soluble in water and forms a colloidal solution. Its tendency to absorb iodine is slight; it is degraded by beta-amylase only to the extent of about 55 percent, and it does not produce a V-type X-ray pattern. Amylopectin has a globular shape, showing increased dilation and consequent higher viscosity in most solvents (19,49).

The Nature of Starch Gelatinization

Native starch granules are relatively insoluble in cold water. However, when heated in the presence of water, the granules become increasingly soluble.

When an aqueous suspension of granular starch is heated, it is presumed that the more amorphous regions undergo progressive hydration and swelling, thus forming an expanded network which gives the swollen granule its elastic character and which is responsible for most of the viscous qualities of a cooked starch paste (35). It should be pointed out, however, that starch paste is a heterogeneous system, each constituent of which may react differently toward changes in the conditions of measured viscosity (8).

Heating starch in the presence of water opens the micellar bundles, thereby allowing water to penetrate into the granules (10). Sandstedt (46) reported that approximately ten or more times the weight of starch in water was absorbed during the gelatinization process. Although the micelles are disrupted during swelling, there is a persistent network that continues to hold the granule together even though greatly expanded. This network may be broken by supplying high levels of energy, such as by autoclaving a dilute starch suspension. The beginning of a significant swelling of individual corn and wheat starch granules commences at 50°C. Although gelatinization may not be in evidence until 65°C. is reached, gelatinization is rapid and may be completed at 67.5°C. The process is dependent on the source of starch (4,26,44). The mean temperature of complete swelling of the individual granules of corn starch was reported by Arzickowski (3) to be 60.9°C. plus or minus 0.10°C. Gelatinization of corn starch begins at 64°C. and continues over a range of 80°C., the shape of the curve resembling the curve for tapioca starch (22).

The short chain amylose molecules were dissolved out of the granule. Generally, the large granules of any starch gelatinize at a lower temperature than the smaller ones (31). The difference in gelatinization of the granules, however, takes place over a range of temperature of about 10°C. (30,48). With the aid of polarographic and amperometric investigations, Taufel and coworkers (52) found that smaller granules dissolve more slowly than the larger granules in spite of the larger specific surface. He concluded that the structure of the large granules was less compact.

McMasters (36) stated that the bright segments within the four arms of the Maltese cross decreased in size and eventually disappear entirely. They usually persisted longer at the periphery of the granule. When all birefringence had been lost, the granule was said to be gelatinized. MacMasters (36) also observed that birefringence reappeared in the outer portion of the gelatinized granule upon the addition of iodine solution. This occurred with starch which had been heated in water to 10 or 15°C. above the temperature at which birefringence was completely lost. These observations suggested that disorganization of the starch granule occurring during gelatinization was incomplete and that some organization was persistent even where birefringence of the granule was no longer microscopically observable.

Gel formation of a starch is related to the hydration capacity of the starch (15,32). Starches derived from various sources differ widely in hydration capacity. Even starches from different wheat varieties vary somewhat in hydration capacity,

but not nearly so much as the values for the relative viscosities of the respective solutions would indicate. Continued heating of gelatinized starch pastes produces a rapid decrease in the hydration capacity. This change is much more rapid for potato starch pastes than for wheat starch pastes (17).

Starches which have predominantly linear fractions, such as wrinkled pea, gelatinize with great difficulty and do not dissolve even after prolonged autoclaving. At the opposite end of the scale, the absence of the linear fraction such as in waxy maize, causes the granule to swell rapidly and disintegrate with continued cooking. Between these extremes are the intermediate gelatinization behavior of corn and wheat starch. These differences are due largely to the content of the linear fraction and the micellar organization within the granule (10).

There is some disagreement on the temperatures at which different starches gelatinize because of the pre-treatment of the starch, difference in varieties of the same species, and also because of different subjective interpretations of the gelatinization point (1,43).

When starch granules are heated in an excess of water, the degree of swelling appears to be a function of temperature. If insufficient water is present, the swelling is greatly inhibited (42). The optimum swelling of the granule is dependent upon an adequate supply of water. Adequate adjustment of available water and the rate of heating may be sufficient to maintain complete control over starch gelatinization.

Anker and Geddes (2), citing Katz's studies of the viscosity changes which occur in corn and potato starch on heating and stirring, concluded that the heat gelatinization curve was a result of two opposing factors: the progressive swelling and hydration of the starch granule which increases viscosity and the breakdown of the granule which decreases viscosity.

Kawamura and Takahashi (29) reported that gelatinization was influenced more by temperature than by heating time. The rise of temperature from 65°C. to 80°C. had great influence on the extent of swelling measured at 90°C. Halick et al. (18) reported that swelling of the starch granule began well before the effect of increased viscosity became apparent. He showed that if the rice flour-to-water ratio was increased (100 grams of rice flour and 400 mls. water), the rise in viscosity was quite abrupt and the point of departure of the curve from the base line could be taken as a measure of gelatinization temperature. According to Anker and Geddes (2), the temperature of transition and paste temperature at maximum viscosity decreased with an increase in starch concentration. They concluded that starch swelling was a first order reaction process and that granule disintegration was a second or higher order process. It was shown by Anker and Geddes (2) that with wheat starch, the first indications of swelling are related to the temperature and the concentration of the starch suspension. The higher the starch concentration is, the lower the temperature at which swelling first occurs. Where a 7 percent wheat starch suspension was gelatinized in water, no indications of swelling were apparent until approximately 86°C. was reached,

as judged by the Brabender Amylograph. From 86°C. to a peak at 93°C., the viscosity increased rapidly, thereafter decreasing because of the mechanical action of stirring on the swollen granule. When a 10 percent wheat starch suspension was gelatinized, the first indication of swelling was evident at 71°C. The peak viscosity occurred at a slightly higher temperature. There was evidence of greater mechanical breakdown of granules with higher starch concentrations. With 10 percent concentration, swelling of the granules occurred from 71°C. to about 93°C., or a range of 22°C. Any further increase in starch concentration resulted in a viscosity greater than could be measured by the Amylograph. It was not possible to demonstrate two stages of gelatinization for wheat starch at concentrations of 10 percent or higher of starch. Two stages were observed by Katz and Rientsma (27) for wheat starch by means of the Roentgen spectrum.

The changes of the structure of the starch granule on heating in water takes place in three discernible phases. During the first phase, water is slowly and reversibly taken up, and limited swelling occurs. The viscosity of the suspension does not increase noticeably. The granule retains its characteristic appearance and birefringence, and upon cooling and drying, no obvious changes can be observed. At approximately 65°C. (the exact temperature being a characteristic of the particular variety of starch) the second phase of swelling starts. The granule suddenly swells, increasing many times in size, absorbing a large quantity of water, and losing its birefringence rapidly. The second phase of swelling is also marked by a rapid rise in the viscosity of the

starch suspension, and the granules upon cooling are altered in appearance, most of them having lost their structure and birefringence. A small amount of the starch has become solubilized. During the third phase of swelling at elevated temperatures, the granules become almost formless sacs and the more soluble part of the starch is leached out (30).

When the granule is heated, the Maltese cross gradually enlarges at the intersection of its arms, i.e., at the hilum of the granule (36). According to Hollo et al. (23), this factor, reported by MacMasters and others, has a bearing in the process of paste formation of the starch. The indication of a temperature of gelatinization has a meaning only if conditions of the measurements are specified. Some of the factors studied by Hollo et al. (23) were moisture, H-ion concentration, dextrinization, and the presence of enzymes.

An interesting practical aspect of starch gelatinization, as related to bread baking, was reported by Betchel (6). Betchel reported that the gluten bound nearly all the water, and the starch bound almost none until the temperature was raised to 125°F. After that temperature, gluten lost its water-holding ability, and starch began to absorb water readily. As the temperature was progressively raised, more water was absorbed by the starch, causing gelatinization. The starch granules did not swell to their fullest extent because of the limited amount of water present.

The characteristic gelatinization properties of starches have been applied to pre-gelatinized starches used as food

thickening agents and stabilizers (53). Products made with pregelatinized starch are usually more uniform and stable to storage and freezing. They offer a considerable saving of production time (13).

Degradation of the Starch Molecules by Beta-amylase

Much study has been devoted to the degradation of the starch fractions by beta-amylase.

Before the enzyme can act appreciably on the starch, it must be gelatinized and its internal structure disorganized so that beta-amylase has the open-chain and reducing ends available to its action. Beta-amylase (M.W. = 150,000) acts on the nonreducing terminal ends of either the linear or the branched fractions, successively splitting off maltose until its action is stopped by a branch point or other anomalous linkage (49). The fission products of amylose are maltose and maltotriose, and those of amylopectin are maltose and beta-amylase limit dextrin. Amylose dextrins (chain length 16-30) may be hydrolyzed by soybean and crystallized sweet potato beta-amylase (24,25). Preece and Shadaksharaswany (41), using soluble starch as the substrate, reported that sugar production (assessed by the ferricyanide titration) by beta-amylase shows a linear relationship with time up to 15 percent hydrolysis. As soon as beta-amylase is mixed with starch, there is an immediate increase in optical activity which is ascribed to the formation of an enzyme-substrate complex (7). Cleveland and Kerr (9) stated that when the enzyme makes contact with an amylose molecule, this chain is hydrolyzed to

maltose before the enzyme attacks another chain. This was shown by hydrolysis of amylose with a limited amount of beta-amylase. Only sugars and unconverted residues which have nearly the same average size as the original sample were obtained. Bailey and Whelan (5) reported that the action pattern of beta-amylase was always intermediate between single and multichain.

The single mechanism can be explained by the reaction:

$$E + Sn \rightleftharpoons (E + Sn) \rightleftharpoons G_2 + (ESn - 2) \rightleftharpoons 2G_2 + (ESn - 4) \text{ etc.}$$

The multichain mechanism is explained as follows:

$$E + Sn \rightleftharpoons (ESn) \rightleftharpoons E + Sn - 2 + G_2$$

Where E = enzyme, S = substrate, n = number of glucose units, and G = glucose.

Their results showed that the enzyme removes several maltose units during each encounter with a substrate molecule. It was suggested that these changes in the pattern of action can be explained by visualizing the enzyme having several active centers or that multiple reactions can occur at a single active center. Schoch (47) reported that beta-amylase must come into exact juxtaposition with the appropriate alpha 1-4 linkage, which it then splits by contact catalysis. It is calculated that one mole of crystalline malt beta-amylase will hydrolyze 250,000 glucosidic linkages per minute. If the starch paste is kept for a long time, starch tends to retrograde and in this form amylolysis is greatly retarded (37).

Starch Retrogradation

If a starch paste is prepared and left standing at room temperature, the solubility equilibrium shifts so that the starch loses solubility and is less hydrated. This tendency has been termed retrogradation and is exemplified by increased opacity and syneresis occurring during aging of starch pastes (27,28). Retrogradation may be considered as the reverse of swelling. The individual molecules in the paste come together to form a large insoluble aggregate with an internal structure similar to that of the original granule. The amylose fraction retrogrades rapidly because the chain molecules are flexible and can readily orient to form crystallites. Branched chain molecules stay in solution much longer. Waxy starch pastes retrograde slowly (21).

Khim (33) has defined retrogradation as those changes in the physico-chemical state of hydrophilic colloids that take place in the solutions, pastes, and gels on aging. The retrograded portion is the coagulum separated out in the clear liquid which contains the un-retrograded portion. The retrograded starch consists of shapeless masses, apparently composed of small, finely-divided granular particles. They do not show double refraction in polarized light and may be readily redissolved by heating at approximately 90°C.

Retrogradation is retarded by agents which tend to disperse the starch, and is aided by those agents that coagulate starch. Dilute starch pastes or solutions of low starch concentration retrograde to form a precipitate, whereas heavy pastes gel, retrograde, and undergo syneresis. At greater concentrations, starch

is deposited until an equilibrium is reached. The extent of retrogradation appears to be governed by the molecular weight of the fraction and by the colloidal structure (34).

MATERIALS AND METHODS

Five cereal starches including wheat, corn, waxy corn, non-glutinous sorghum, and German rice were analyzed for beta-amylase susceptibility and maximum hot-paste viscosity changes following pre-heat treatment at two temperatures, 100°C. and 200°C., using five different moisture levels.

Commercial wheat starch (Starbake) was used as a source of wheat starch. German rice and unmodified corn starches were obtained from Corn Products Co., Argo, Illinois. Waxy corn starch was obtained from American Maize Products. Non-glutinous sorghum starch was obtained from the Department of Flour and Feed Milling Industries, Kansas State University.

The work was divided into two parts according to the temperatures used. The first part comprised the beta-amylolysis and hot paste viscosity determinations after pre-treatment of the starch at 100°C. The second part comprised these same determinations after pre-treatment of the starch at 200°C.

Part I

Beta-amylolysis. In preparation of the starches for the beta-amylolysis determination, 1.1 grams of starch was mixed thoroughly with distilled water to provide starches with moisture contents as follows: 29, 38, 52, 68, and 99 percent. The

starches were then heated in an autoclave at 100°C. for five minutes. After autoclaving and cooling, additional water was added, and the paste was passed through a silk gauze (G.G.54) in order to disperse the suspension. The resulting starch suspensions were diluted to 100 ml. with water and blended for 15 seconds in a Waring blendor to insure uniform dispersion. It should be pointed out here that although the blending of the starch provided a uniform suspension, its use did not increase the starch susceptibility to beta-amylase action in a consistent way as shall be demonstrated in the discussion of results.

Forty ml. of these suspensions were pipetted into 125-ml. flasks, five ml. of acetic acid-sodium acetate buffer (pH 4.60) were added, and the suspension was placed in a water bath at 30°C. Five ml. of a solution containing 4 mg. of beta-amylase[1] were added to initiate the beta-amylolysis.

Five ml. of the starch-enzyme suspension were vacuum filtered (medium-size sintered glass filters were used) immediately after the addition of the enzyme. This sample was used as the "zero" time sample. Three ml. of the filtrate were pipetted into test tubes containing 10 ml. of 0.1 N solution of alkaline potassium ferricyanide. The test tubes were immersed in a boiling water bath for 20 minutes. At the end of the 20-minute boiling time, the tubes were immersed in a cold water bath and brought to room temperature. The contents of the test tubes were transferred to 125-ml. flasks with the aid of 25 ml. of acetic

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[1] Wallerstein Company, New York, New York.

acid-salt solution. The non-reduced ferricyanide was titrated with 0.1 N sodium thiosulfate solution, using 1 ml. of potassium iodide-iodine soluble starch reagent as the indicator. A sample of the starch-enzyme suspension was taken every hour for three hours.

Hot Paste Viscosity. The hot paste viscosity determinations were made by using 45 grams of the starch (14% M.B.) and sufficient water to obtain the same moisture content as employed in the beta-amylolysis determinations. After pre-heating the starches, they were suspended in 400 ml. of disodium phosphate-citric acid buffer (pH 5.35). The maximum hot-paste viscosity was recorded with the Brabender amylograph by heating at the rate of 1.5°C. per minute. Maximum viscosity was taken as the pertinent measure of paste formation.

Part II

Beta-amylolysis. The starch suspensions for the beta-amylolysis determination for pre-heat treatment at 200°F. were prepared in exactly the same manner as for Part I under beta-amylolysis. Ten grams of starch were used instead of 1.1 grams and the amount of water adjusted to obtain the same moisture levels in the suspensions as previously specified.

Stainless steel tubes (6½ inches long and 0.5 inch in internal diameter) hermetically sealed by means of a steel cap with a lead gasket were used as containers for the pre-heat treatment of the starches. The heating at 200°C. was done in a Reed reel

oven[1] for five minutes. At the end of the heating time, the starches were transferred from the tubes and treated as described under Part I for beta-amylolysis. Because of the limited volume capacity of the tubes, 0.25 gram of starch was used for the 99.01 percent moisture level sample, and the water adjusted accordingly to obtain the desired moisture content.

Hot Paste Viscosity. The same stainless steel tubes as described above were used for the pre-heat treatment of the starches for this determination, except that the 45 grams of the starch-water mixture were equally divided between five tubes and heated at 200°C. in the Reed-reel oven for five minutes. At the end of the heating period the tubes were allowed to cool at room temperature. The starch paste was then transferred from the tubes and treated as described under Part I for hot-paste viscosity.

A control was prepared by mixing 45 grams of the raw starch with 400 ml. of disodium phosphate-citric acid buffer, and the maximum hot-paste viscosity determined with the Brabender Amylograph.

All these determinations were undertaken in order to measure the total effect of the moisture-temperature relationship on the starch-water system as a whole and not on each granule as an entity. It was recognized at the beginning that uniform swelling of all granules in the pre-heat treatment would not be attained in the system employed.

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[1] Serial No. HL 8 304. Bakers Engineering and Equipment Company, Kansas City, Kansas.

RESULTS AND DISCUSSION

The starches varied in appearance after the pre-heat treatment. The two starches with the lower moisture levels, 29 and 38 percent moisture, had a consistency similar to that of an agglutinated powder. The starch gels made with 52 and 68 percent moisture were hard, elastic pastes with a more or less dry core in the center. The starch gels made with 99 percent moisture were fluid. These properties of the starch gels existed for both the 100° and 200°C. pre-heat treatment.

The hard, dry core in the center of the starch gels made with 52 and 68 percent moisture levels was due to the uneven distribution of the water throughout the starch mass. As the heat penetrated from the outside of the container, the granules close to the walls of the container swelled and absorbed the water, thus removing the water from the center of the starch mass. This effect caused a non-uniform wetting of the granules in the gel throughout the system with only a percentage of the granules being fully gelatinized. The technique employed here was selected to measure the total moisture-temperature relationship on the entire system. This approach was taken in view of the practical applications of such findings to commercial food processing.

Difficulty in dispersing the starch gels was experienced early in this investigation. The pastes made with moisture levels of 52 to 68 percent were tough, elastic gels which defied dispersion in water or buffer by shaking or gentle stirring. It was felt that a uniform dispersion of the starch gel was necessary for

representative sampling as well as for having the starch readily available to the beta-amylase. After investigating several techniques, including the use of inert materials such as celite, glass beads, and sand to reduce the gel strength, without success, the use of the Waring blendor to disperse the starch was used. In order to determine the effect of this procedure on the availability of the starch to beta-amylase, a series of wheat starch gels was prepared varying in moisture. These gels were analyzed for starch availability to beta-amylase with and without the use of the Waring blendor to disperse the starch. The starch gels partially dispersed in water were blended for but 15 seconds. The results of this study are presented in Table 1. The use of the Waring blendor to disperse the resulting pastes after pre-heat treatment showed no consistent effects on the availability of the starches to beta-amylase.

Table 1. Effect of the Waring blendor on the availability of pre-heated wheat starch to beta-amylase.

Moisture level (%)	Treatment	Digestion time (hr.) 1	2	3
		Maltose equivalents (mg.)		
29	Blended	0.3	0.6	0.6
	Not blended	0.8	1.1	1.1
38	Blended	1.1	1.9	2.4
	Not blended	1.1	2.9	3.9
52	Blended	6.5	7.9	8.6
	Not blended	6.3	7.8	7.8
68	Blended	8.6	8.9	9.6
	Not blended	6.5	7.9	9.5

Availability of starch to beta-amylolysis increased with each increment of moisture. The effect of the moisture-heat treatment was characteristic for each starch as illustrated by the data in Tables 2 to 6 and Figs. 1 to 5. Availability of the starch to beta-amylase increased with each increment of moisture whether heated at 100 or 200°C. However, heating at the elevated temperature caused a significant decrease in availability of starch at any given moisture level. No data are presented for moisture levels below 29 percent because at lower moisture levels, the effect of the heat-moisture treatment could not be demonstrated by the technics employed. The results of pre-heat treatment of wheat starch are shown in Tables 2 and 3 and Figs. 1 and 2.

Table 2. Maltose production by beta-amylolysis of wheat starch after heating at various temperatures and at different moisture levels.

Moisture level %	Pre-heat treatment °C.	Digestion time (hr.) 1	2	3	Maximum availability* %
		Maltose equivalents (mg.)			
29	100	0.30	0.60	0.60	6.10
	200	0.00	0.00	0.30	4.23
38	100	1.10	1.90	2.40	24.70
	200	0.00	0.20	0.50	7.05
52	100	6.50	7.90	8.60	88.60
	200	1.50	2.10	2.60	36.60
68	100	8.60	8.90	9.60	97.00
	200	3.90	5.20	5.50	77.50
99	100	8.60	9.20	9.70	100.00
	200	4.10	5.50	7.10	100.00

* Expressed as percent of available starch after three hours digestion assuming 99 percent moisture starch as 100 percent available to beta-amylase.

Table 3. Hot peak viscosities of the starches.

Moisture level %	Pre-heat treatment °C.	Starch				
		Wheat	Non-glutinous sorghum	Waxy corn	Unmodified corn	German rice
		B.U.				
Control		620	900	1000	900	1000
29	100	520	720	1000	700	980
	200	480	680	1000	700	950
38	100	390	550	1000	565	975
	200	310	490	1000	520	947
52	100	341	415	470	338	960
	200	237	280	1000	180	580
68	100	290	220	200	315	840
	200	225	175	845	170	475

Wheat Starch

The hot paste viscosity is directly affected by the degree of internal disorganization of the starch granule which in turn is affected by temperature, available moisture, and amylose-amylopectin ratio (10,30,29,6). Pre-heating of the starches at 100°C. resulted in a decrease in the hot-paste viscosity as the available moisture was increased during pre-gelatinization. The hot-paste viscosity of the starches after pre-treatment at 200°C. was lower than that given by pre-treatment at 100°C.

As the moisture level was increased with heating to 100°C., the starch became increasingly available to beta-amylase. However, the increase was not linear for all levels investigated. There was a difference in available starch of 63 percent between the moisture contents of 38 and 52 percent; but, between the

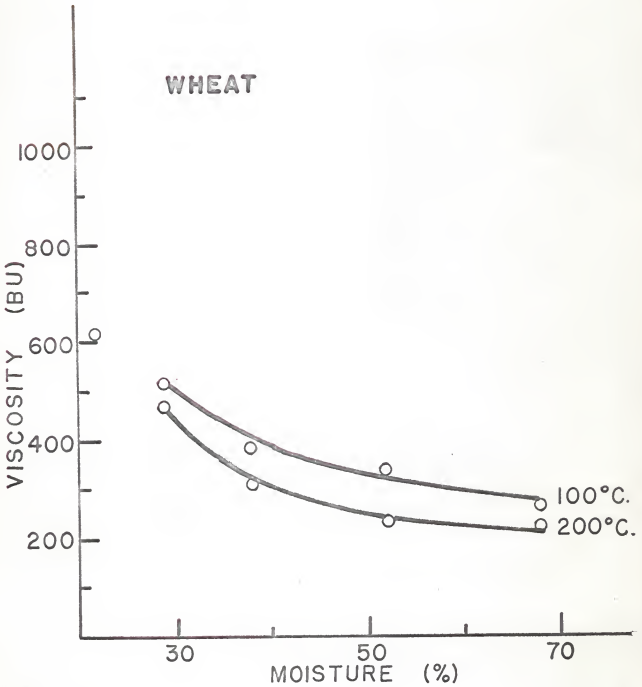

Fig. 1. Effect of moisture and pre-heat treatment on hot-paste viscosity.

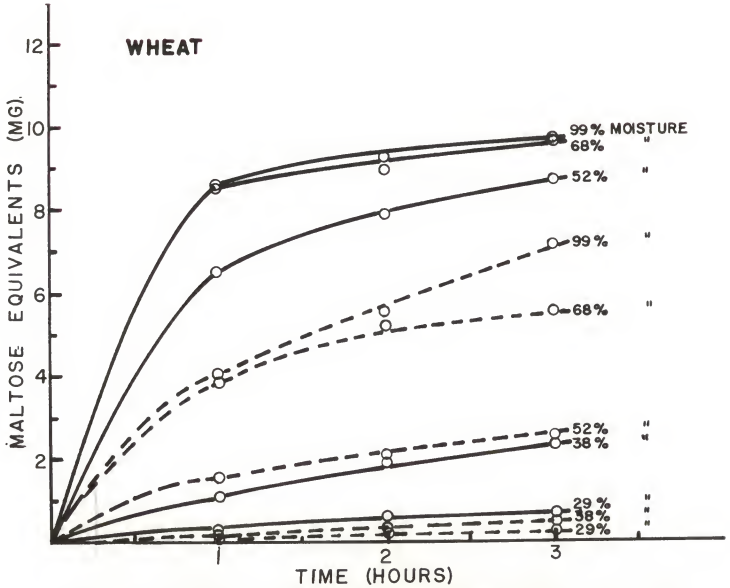

Fig. 2. Effect of moisture and heating at 100°C. (_____) and 200°C. (-----) on beta-amylolysis.

68 and 99 percent moisture levels, the difference was only 3 percent in available starch as measured by maltose produced after three hours of enzyme digestion.

Compared to heating of the starch at 100°C., heating at 200°C. resulted in a decrease in availability of the starch to beta-amylase at all moisture levels. The availability of the starch at the 99 percent moisture level was 37.3 percent lower than that given by the same moisture level at 100°C.

Wheat starch decreased in hot-peak viscosity from 520 B.U. at 29 percent moisture level to 290 B.U. at the 68 percent moisture level. Pre-gelatinizing the starch at 200°C. gave lower peak viscosity values and they were almost parallel to that of the 100°C. treatment. Results are expressed in Table 3 and Fig. 2.

The beta-amylolysis data indicate that at a moisture level of approximately 65 to 70 percent, practically all of the starch granules were gelatinized and therefore generally available to beta-amylase. Furthermore, these data indicate that the degree of gelatinization is dependent on moisture levels.

Apparently, as the temperature is increased when water is limited, the starch molecules are not expanded and therefore are less vulnerable to the action of the beta-amylase. As the moisture becomes even more limited, the starch molecules are less extended and therefore even less available to attack by beta-amylase.

The Amylograph data also seem to support this postulate because pre-treatment of the starch at 200°C. resulted in an even

lower hot-paste viscosity at higher moisture levels. This suggests that the formation of the new internal bonds prevents the starch molecules from forming the expanded network which is responsible for the higher viscosity of gelatinized starch pastes.

Unmodified Corn Starch

The effects of pre-heat treatment of corn starch are shown in Tables 3 and 4 and Figs. 3 and 4.

Table 4. Maltose production by beta-amylolysis of unmodified corn starch after heating at various temperatures and at different moisture levels.

Moisture level	Pre-heat treatment	Digestion time (hr.)			Maximum availability*
		1	2	3	
%	°C.	Maltose equivalents (mg.)			%
29	100	0.43	0.66	0.66	5.08
	200	0.00	0.30	0.60	8.45
38	100	1.23	2.00	3.53	28.46
	200	0.00	0.50	1.00	14.05
52	100	6.06	7.65	7.90	63.54
	200	1.60	3.10	3.60	47.90
68	100	7.93	8.66	9.33	75.20
	200	2.90	4.20	4.90	65.40
99	100	10.06	11.93	12.40	100.00
	200	5.80	6.20	7.50	100.00

* Expressed as percent of available starch after three hours digestion assuring 99 percent moisture starch as 100 percent available to beta-amylase.

The unmodified corn starch gave higher maltose values than the wheat starch when gelatinized at the 99 percent moisture level. This occurred at both temperatures. It would appear that

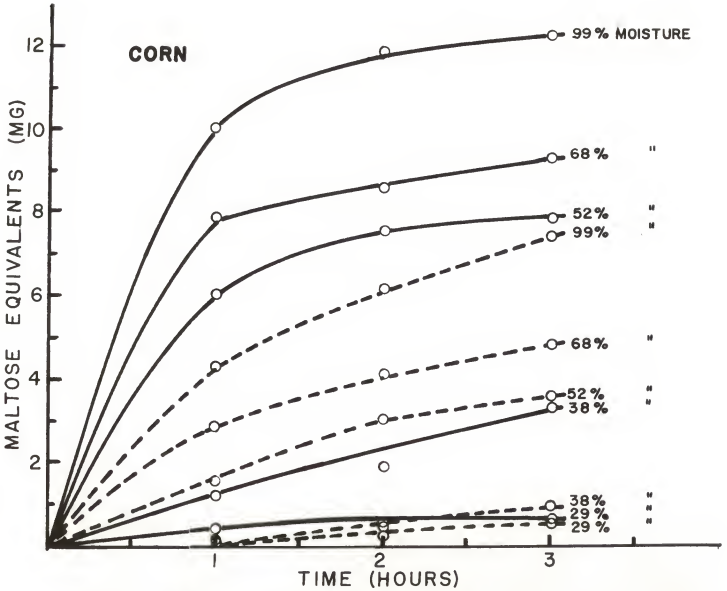

Fig. 3. Effect of moisture and heating at 100°C. (———) and 200°C. (-----) on beta-amylolysis.

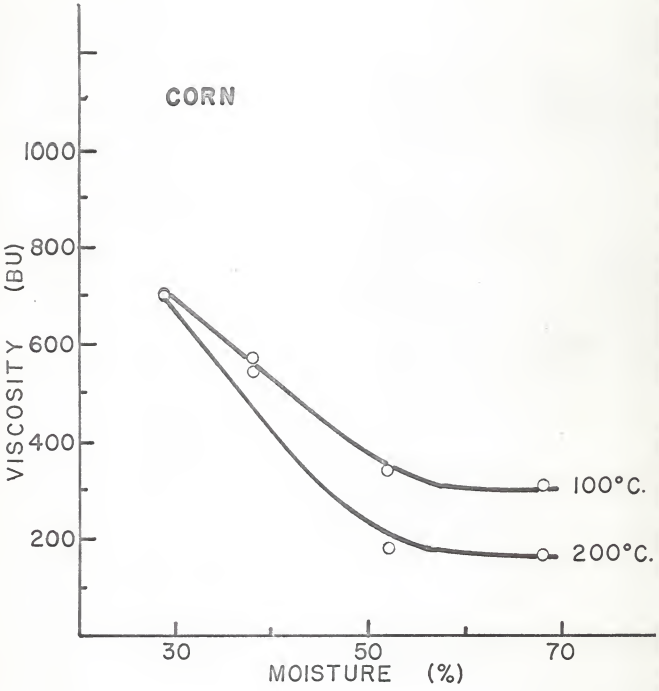

Fig. 4. Effect of moisture and pre-heat treatment on hot-paste viscosity.

corn starch required more water than other starches for an equivalent amount of gelatinization. The maltose production of the different concentrations at 100°C. varied considerably. The smallest difference existed between the 52 percent moisture and the 68 percent moisture levels. The 29 percent moisture starch showed a slight increase between the first and the second hour but showed no increase between the second and the third hour. The other moisture levels tended to show a steady increase in maltose production during the three hours of digestion. As with wheat starch, pre-heat treatment at 200°C. decreased the availability to beta-amylase. In this case the difference for the 99 percent moisture levels at 100°C. and 200°C. was 39.5 percent. The curves for all the moisture levels tended to increase throughout the three hours of digestion.

Unmodified corn starch exhibited a very marked decrease in hot-paste viscosity between the 29 and the 52 percent moisture levels for both temperature treatments. However, the maximum viscosity at 200°C. pre-treatment was lower. Beyond a moisture level of 52 percent there was little effect of the elevated temperature on reduction of the viscosity.

Corn and wheat starch behaved much in the same way although the former gave higher maltose values and lower viscosity. The general trend was more or less similar, indicating that the molecular arrangement of the starches was similar. Some species variation was observed and this is in agreement with the literature (1,10,43).

Waxy Corn Starch

The effects of pre-heat treatment of waxy corn starch are reported in Tables 3 and 5 and Figs. 5 and 6. The three highest moisture levels of this starch gave the highest maltose production of all starches when heated at 100°C. The 99 percent moisture level gave a maltose value of 12.70 mg. maltose equivalents after three hours of digestion. The 68 percent moisture level had 92.1 percent of the starch available, and 52 percent moisture level had 87.5 percent of the starch available to beta-amylase digestion. The 29 percent moisture level showed no increase after the second hour of digestion. After the 200°C. pre-treatment, the 99 percent moisture level had a 50.3 percent lower starch availability than the same moisture level treatment at 100°C. All other moisture levels showed marked differences, with only the 99 and the 68 percent moisture levels showing increased amounts of available starch. The starch treated at 200°C. gave lower maltose values than those treated at 100°C.

Waxy corn starch was the only starch that gave a higher hot-paste viscosity for heat treatment at 200°C. as compared to 100°C. The treatment at 100°C. caused a large drop in hot-paste with a moisture level of 68 percent compared to 38 percent. The 200°C. pre-treatment gave a straight line at 1000 B. U. until the 52 percent moisture level was attained. After that point it dropped to 845 B. U. with pre-treatment with 68 percent moisture.

These results suggest that the molecular bonding in the granule of waxy corn starch is weaker than that of the other

Table 5. Maltose production by beta-amylolysis of waxy corn starch after heating at various temperatures and at different moisture levels.

Moisture level %	Pre-heat treatment °C.	Digestion time (hr.) 1	2	3	Maximum availability* %
		Maltose equivalents (mg.)			
29	100	0.30	0.60	0.60	4.70
	200	0.00	0.20	0.30	5.18
38	100	3.20	3.70	4.20	33.00
	200	0.50	0.80	0.80	13.80
52	100	9.90	10.50	10.90	85.70
	200	3.10	3.60	3.70	63.90
68	100	10.70	11.30	11.70	92.10
	200	3.10	4.20	5.20	89.90
99	100	12.20	12.50	12.70	100.00
	200	4.50	4.70	5.80	100.00

* Expressed as percent of available starch after three hours digestion assuring 99 percent moisture starch as 100 percent available to beta-amylase.

starches used in this study. As a consequence, after gelatinization, beta-amylase can penetrate and act on the starch much easier. The second suggestion of these data is that the large amylopectin content of this starch promoted the formation of large numbers of cross bonds when the starch was heated at 200°C. This higher degree of bonding prevented beta-amylase from attacking the starch as extensively as it was when heated at 100°C. The hot-paste viscosity remained high when heated to 200°C., in spite of greater bond formation, because of the extensive network resulting from the high content of amylopectin.

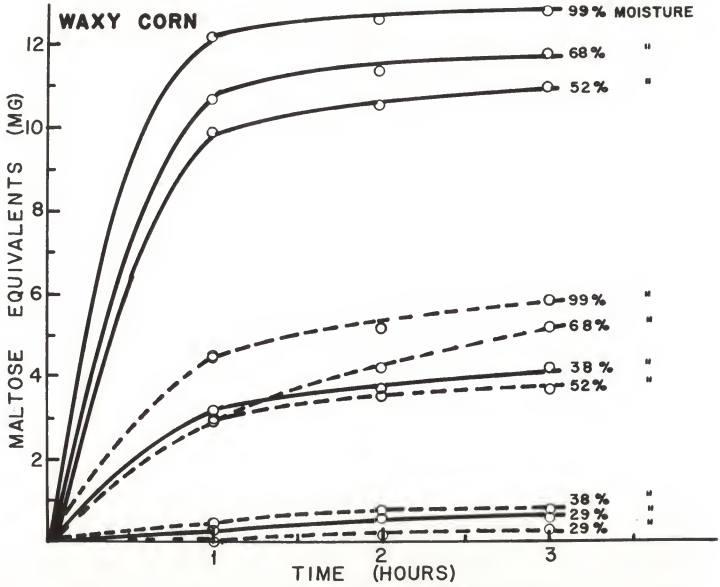

Fig. 5. Effect of moisture and heating at 100°C. (———) and 200°C. (-----) on beta-amylolysis.

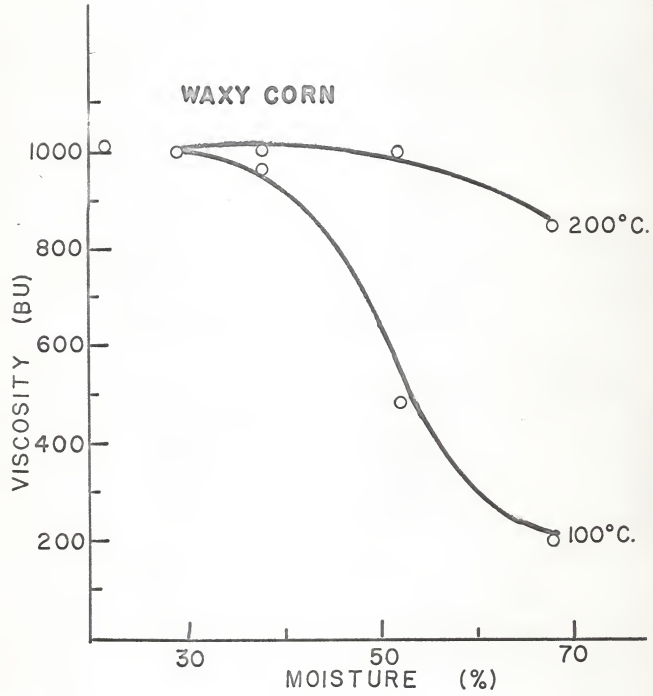

Fig. 6. Effect of moisture and pre-heat treatment on hot-paste viscosity.

Non-glutinous Sorghum Starch

The effects of pre-treatment of non-glutinous sorghum starch are reported in Tables 3 and 6 and Figs. 7 and 8. The results obtained after pre-treatment of non-glutinous sorghum starch at 100°C. were similar to those obtained for wheat starch although a slight increase in maltose production was observed for the non-glutinous sorghum starch. The beta-amylolysis of the non-glutinous sorghum starch demonstrated an important difference in moisture requirement for gelatinization. At moisture levels of 68 and 52 percent, maltose production was 83.76 and 72.27 percent of the 99 percent moisture level while the two lower moisture levels gave maltose equivalents of 26.73 and 8.91 percent of the 99 percent moisture levels, respectively.

Pre-heat treatment at 200°C. gave lower starch availability at all moisture levels. A difference of 32.77 percent was found between the 99 percent moisture levels treated at 100° and 200°C. Starch availability increased at all moisture levels with the exception of the 29 and the 38 percent moisture levels which gave almost identical results. The maximum hot-paste viscosity reduced steadily as the moisture level increased for the pre-heat treatment at 100°C. Pre-treatment at 200°C. lowered the maximum hot-paste viscosity values but the curve was almost parallel to that for the 100°C. pre-heat treatment. Both the amylograph and the beta-amylolysis data showed that the behavior of non-glutinous sorghum starch was very similar to that of wheat starch, suggesting a comparable internal arrangement of the granules.

Table 6. Maltose production by beta-amylolysis of non-glutinous sorghum starch after heating at various temperatures and at different moisture levels.

Moisture level %	Pre-heat treatment °C.	Digestion time (hr.) 1	2	3	Maximum availability* %
		Maltose equivalents (mg.)			
29	100	0.30	0.90	0.90	8.91
	200	0.00	0.80	0.80	12.15
38	100	1.73	2.50	2.70	26.73
	200	0.20	0.80	0.80	12.15
52	100	7.80	8.53	9.36	88.07
	200	1.80	2.80	3.70	56.90
68	100	8.96	9.86	10.06	94.50
	200	1.90	3.10	3.90	60.00
99	100	9.46	10.20	10.63	100.00
	200	5.00	5.80	6.50	100.00

* Expressed as percent of available starch after three hours digestion assuring 99 percent moisture starch as 100 percent available to beta-amylase.

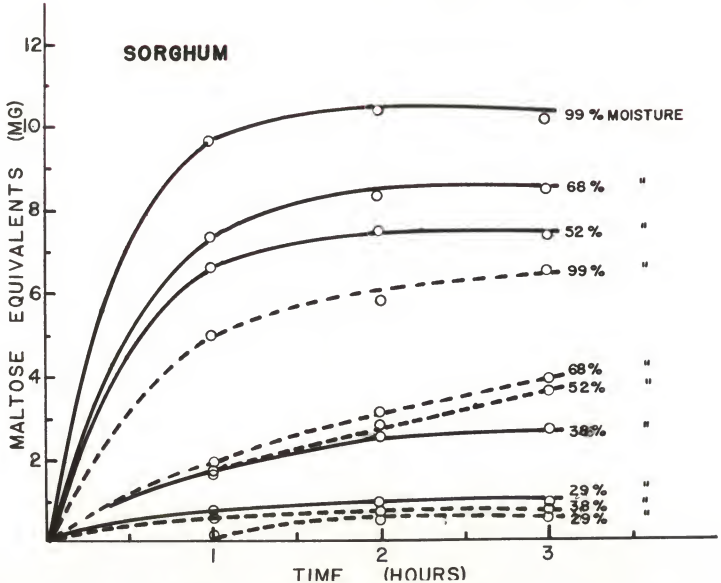

Fig. 7. Effect of moisture and heating at 100°C. (———) and 200°C. (-----) on beta-amylolysis.

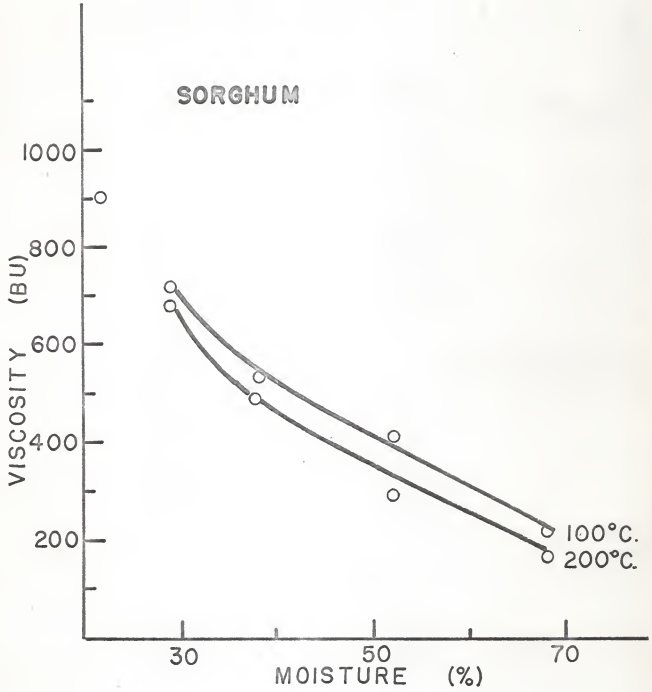

Fig. 8. Effect of moisture and pre-heat treatment on hot-paste viscosity.

German Rice Starch

The effect of pre-treatment of rice starch is reported in Tables 3 and 7 and Figs. 9 and 10. Starch availability after pre-heat treatment at 100°C. was large if sufficient water was present. At low moisture levels, while the starch availability was low, it was still higher than for other starches when compared at the same levels. Pre-heat treatment at 200°C. reduced the starch availability at the higher moisture levels but had little, if any, effect at the low moisture levels. This suggests that moisture has a distinct effect on bonding between molecules during the gelatinization process. With a starch such as German rice, bond formation after pre-heating was not high enough to inhibit the action of beta-amylase. This view is also supported by the maximum hot-paste viscosity. The hot-paste viscosity reduction as moisture increased was significant.

The data presented in this research work suggested that pre-heating the starch at 100°C. or 200°C., in the presence of water, opens the micellar bundles, causing the rupture of the original structure of the starch granule. The structural disorganization leads to the exposure of the open ends of the starch molecule, thus causing them to become vulnerable to the attack by beta-amylase. In addition, pre-heating the starches at 200°C. caused the formation of new bonds due to the interaction of starch molecules among themselves. This was particularly evident at the high moisture levels. When the moisture level was below 38 percent, insufficient water was present to cause bond rupture within the granule, thus causing small availability of the starch to beta-amylase.

Table 7. Maltose production by beta-amylolysis of German rice starch after heating at various temperatures and at different moisture levels.

Moisture level %	Pre-heat treatment °C.	Digestion time (hr.) 1	2	3	Maximum availability* %
		Maltose equivalents (mg.)			
29	100	1.60	2.13	2.76	25.90
	200	0.50	0.80	1.00	10.20
38	100	4.50	5.36	6.33	59.40
	200	0.50	0.80	1.10	11.70
52	100	7.80	8.53	9.36	88.07
	200	2.10	2.90	3.60	38.30
68	100	8.96	9.86	10.06	94.50
	200	4.10	6.00	7.30	77.86
99	100	9.46	10.20	10.63	100.00
	200	7.80	8.90	9.40	100.00

* Expressed as percent of available starch after three hours digestion assuring 99 percent moisture starch as 100 percent available to beta-amylase.

These findings are in agreement with other investigators (10,19, 22,30,35,42,46) who have found that starch gelatinization and bond rupture are intimately associated with the presence of water during heating.

Results of this investigation suggest that the amylose-amylopectin ratio of the starch has a direct relation to the effect of moisture on the gelatinization properties. As the amount of amylopectin increased in the ratio, the bonding between starch molecules in the granule became weaker. This conceivably permitted water to penetrate the granule more readily with subsequent swelling. Such granules became more available to beta-amylase action. In those starches having similar amylose-amylopectin

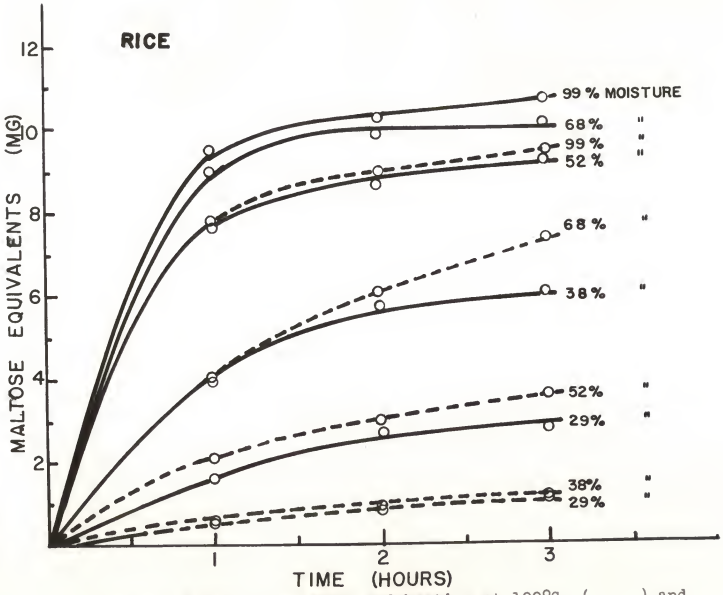

Fig. 9. Effect of moisture and heating at 100°C. (——) and 200°C. (-----) on beta-amylolysis.

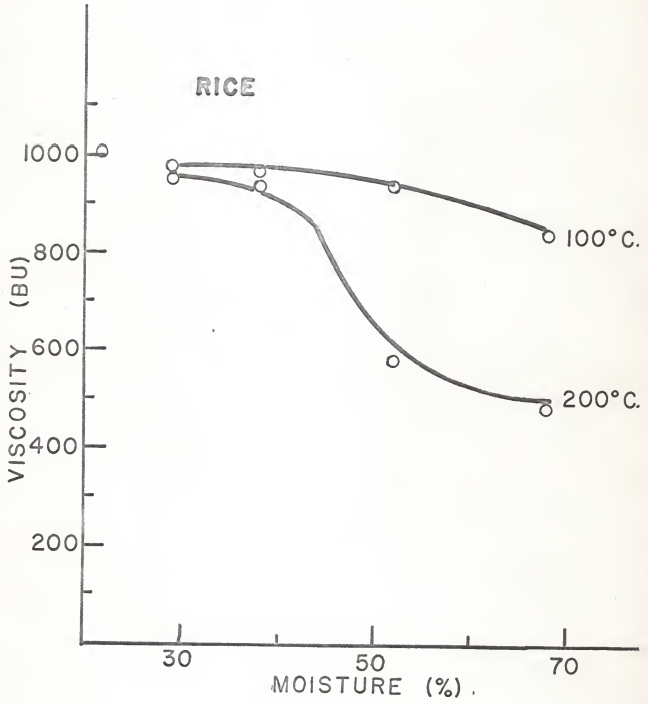

Fig. 10. Effect of moisture and pre-heat treatment on hot-paste viscosity.

ratios such as wheat, corn, and sorghum, the gelatinization characteristics were almost alike but distinctly different from those of the waxy corn and German rice starches.

It is reasonable to assume that pressure developed inside the tubes when starch was heated at 200°C., and that such pressure probably enhanced the formation of new bonds. These starches were less available to beta-amylase action and had a considerably lower hot-paste viscosity.

CONCLUSIONS

An investigation of the effect of moisture and temperature on starch gelatinization has been conducted. Moisture may be a limiting factor in starch gelatinization as shown by the increasing availability of the starch to beta-amylase with an increase in moisture. An increase in moisture resulted in a decrease in maximum hot-paste viscosity. Heating had a greater effect on the starch gelatinization as moisture became more available. With sufficient available moisture, an increase in temperature decreased starch availability to beta-amylase and reduced hot-paste viscosity. An insufficient amount of moisture during heating caused an uneven gelatinization of the starch. The granules closer to the surface absorbed most of the water present, leaving the granules in the center of the system unaltered.

The amount of branching affected the degree of gelatinization at 100°C. and therefore the availability of the starch to enzyme attack. Granules with high amylopectin content such as waxy corn starch appeared to have weaker intermolecular bonds.

Heating the starches at 200°C. in hermetically-sealed containers appeared to cause an intermolecular rearrangement with the formation of new bonds which were responsible for the decrease of the starch availability to beta-amylase and maximum hot-paste viscosity.

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MOISTURE-TEMPERATURE RELATIONSHIP IN STARCH GELATINIZATION

by

GUSTAVO ENRIQUE PEREZ

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AN ABSTRACT OF A MASTER'S THESIS

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The effects of moisture and temperature treatment on gelatinization and pasting properties of the starches from wheat, corn, waxy corn, non-glutinous sorghum, and German rice were investigated. The starches, with different moisture levels, were pre-treated at 100 and 200°C. and beta-amylolysis and hot-paste viscosity measured to follow the changes in gelatinization and pasting properties. Beta-amylolysis determinations were made using the ferricyanide reduction method, and the Brabender Amylograph was used to measure the maximum hot-paste viscosity.

The results indicated that heating at both temperatures had little or no effect on starch gelatinization if insufficient water was present. In general, when the starches were heated at 100°C., the beta-amylase action on the starches increased, and maximum hot-paste viscosity decreased as the moisture was increased. As the temperature was raised from 100 to 200°C., both starch availability to beta-amylase and maximum hot-paste viscosity decreased for all starches except waxy corn. When sufficient available water was present, pre-treatment at 100°C. caused the internal molecular rearrangement of the starches. At 200°C. pre-treatment, the rearrangement of the starch molecules was more pronounced and resulted in decreased starch availability and reduced maximum hot-paste viscosity.